Thermophysical Properties of Solid and Liquid 90Ti-6Al-4V in the Temperature Range 1400 to 2300 K Measured by Millisecond and Microsecond Pulse-Heating Techniques

E. Kaschnitz, and P. Reiter
Österreichisches Giesserei-Institut
Parkstrasse 21
8700 Leoben, Austria

J. L. McClure
Metallurgy Division, National Institute of Standards and Technology
Gaithersburg, Maryland 20899, USA

Author for Correspondence: E. Kaschnitz (kaschnitz.ogi@unileoben.ac.at)

ABSTRACT

Heat capacity and electrical resistivity of 90Ti-6Al-4V in the temperature range 1400 to 2300 K were measured by two pulse-heating systems, operating in the millisecond and microsecond time regimes, respectively. The millisecond-resolution technique is based on resistive self-heating of a tube-shaped specimen from room temperature to melting in less than 500 ms and measuring current through the specimen, voltage drop along a defined portion of the specimen, and temperature of the specimen every 0.5 millisecond. The microsecond-resolution technique is based on the same principle using a rod-shaped specimen, but the heating rate is faster by a factor of 10,000 and data is recorded every 0.5 microsecond.

Due to the rapid heating with the microsecond system, the specimen keeps its shape even in the liquid phase, and measurements were made up to approximately 300 K above the melting temperature. A comparison between the results obtained from the two systems with very different heating rates shows significant differences in the region of the phase transition from (α+β) to β and changes in the melting behavior. The very high heating rate of the microsecond system shifts the solid-solid phase transition to a higher temperature and melting of the specimen occurs with a temperature plateau instead of a temperature interval.

Key words: electrical resistivity; high temperatures; pulse heating; specific heat capacity; titanium alloy (90Ti-6Al-4V); transient techniques.

1. INTRODUCTION

In this paper, application of transient heating techniques (millisecond and microsecond) to the measurements of selected thermophysical properties (specific heat capacity, electrical resistivity) of the alloy 90Ti-6Al-4V in the vicinity of melting is described. The method is based on rapid resistive self-heating of the specimen from room temperature to high temperature in a short time by the passage of an electrical current pulse. Simultaneously, the specimen temperature, the current through the specimen, and the voltage drop across a defined portion of the specimen is measured.

2. EXPERIMENTAL

2.1. Millisecond pulse-heating system

The millisecond pulse-heating system uses heavy truck batteries to supply the current . The current through the tubular specimen is determined by measuring the voltage across a standard resistor in series with the specimen. The voltage across the middle part of the specimen is measured between spring-loaded knife-edge probes. They are mounted to the clamps via a lever-mechanism that allows the probes to move in the axial direction to follow the thermal expansion of the specimen during the experiment. The temperature is measured by means of a high-speed single wavelength pyrometer, targeted at a small sighting hole machined through the wall of the specimen, thereby approximating a blackbody cavity. The target size of the pyrometer is 0.2 mm, the working wavelength of 902 nm is selected by an interference filter with a bandwidth of 20 nm. The radiation

is collected by a silicon PIN photodiode and converted to voltage by precision amplifiers. The pyrometer is calibrated to a tungsten-filament standard lamp, which in turn has been calibrated by the German Calibration Service (DKD). Small corrections are made for the deviation from blackbody conditions (approx. 1.6 K) and for scattered light effects (approx. 2.2 K) [1].

The experimental quantities are recorded simultaneously every 0.5 ms by a data acquisition system with sample-and-hold amplifiers and a full-scale resolution of 16 bits. Details regarding construction of the system is given in [2]. Measurements are taken in the temperature range from 1400 K to 1900 K. At melting, the specimen collapses due to electromagnetic and gravitational forces and the effects of surface tension.

2.2. Microsecond pulse-heating system

The microsecond pulse-heating system uses high-voltage capacitors to supply the current and the current through the rod-shaped specimen is determined using a current transformer. The voltage across the specimen is determined by measuring the current through a high-resistance path connected in parallel across the specimen. The radiance temperature is measured with a high-speed pyrometer, targeted at the surface of the specimen. The working wavelength of the pyrometer is 651 nm with a bandwidth of 30 nm. True temperature was calculated from radiance temperature assuming a constant normal spectral emissivity in the liquid and equal to the value at the beginning of melting. The normal spectral emissivity at beginning of melting was determined from

the measured radiance temperature and the true melting temperature obtained in millisecond-resolution experiments [3]. The pyrometer is calibrated to a tungsten-filament standard lamp, which in turn has been calibrated by the Radiation Division at NIST.

The experimental quantities are recorded simultaneously every 0.2 μs by a four-channel digital oscilloscope with a full-scale resolution of 12 bits. Details regarding construction of the system is given in [4]. Due to the rapid heating with the microsecond system, the specimen keeps its shape even in the liquid phase, and measurements were made up to approximately 300 K above the melting temperature.

3. MEASUREMENTS

3.1. Specimens

The measurements of specific heat capacity and electrical resistivity in the solid range (millisecond experiments) were performed on tubular specimens. The nominal dimensions of the tubes were: length, 75 mm; outside diameter, 9 mm; wall thickness, 1 mm. For the temperature measurements, a rectangular hole (0.5 mm x 1.3 mm) was machined through the wall. The departure from ideal blackbody conditions for the specimen was estimated to be smaller than 1% [5]. A total of 15 measurements on three specimens was made.

The measurements in the liquid range (microsecond experiments) were performed on five wire-shaped specimens with 50 mm in length and 1.6 mm in diameter. Although the tubes and wires were supplied by different manufactures, their chemical composition shows little differences as can be seen in Table I.

3.2. Procedure and Data Reduction

All experiments were performed with the specimen in an argon environment at atmospheric pressure. All temperatures reported in this paper are based on the International Temperature Scale of 1990 [6,7].

Specific heat capacity in the solid range was computed from data taken during the heating period of the millisecond experiments. A correction for power loss of up to 3% at the highest temperatures due to thermal radiation was made using data measured during the cooling of the specimen. From the power balance for the specimen during heating and cooling periods, the specific heat capacity c_P as a function of temperature T can be expressed as

$$c_P(T) = \frac{U(T)\, I(T)}{m\left[\left(\frac{dT}{dt}\right)_h (T) - \left(\frac{dT}{dt}\right)_c (T)\right]} \qquad (1)$$

where U is voltage, I is current, m is effective mass, and dT/dt is the heating rate (subscript h) and cooling rate (subscript c), respectively. Heating and cooling rates are treated as independent variables and are obtained by a polynomial fit of the temperature versus time data for the heating period and by an exponential fit for the cooling period.

Specific heat capacity in the liquid range was calculated from the slope of the enthalpy versus temperature function obtained from the microsecond experiments. Enthalpy as a function of time was computed using the mass of the effective specimen and the absorbed energy obtained by numerically integrating current and voltage over time from the beginning of the heating period. Since temperature is measured with the same time base, enthalpy as a function of temperature was obtained by evaluating both at common times. Because of the speed of the experiments, no correction for heat losses was required.

Electrical resistivity of the specimen was computed by means of voltage, current, cross-section and length. All obtained results are based on room temperature dimensions of the specimens and no correction for thermal expansion is applied. It should be noted that the specimen expands radially and axially in millisecond experiments, but due to the high speed of microsecond experiments, the expansion occurs mostly in the radial direction.

4. RESULTS

4.1. Specific Heat Capacity

The variation of specific heat capacity as a function of temperature in the solid and liquid range is shown in Table II. The data from the millisecond experiments are presented for the solid alloy. The reproducibility of measurements for an individual specimen is 0.6% and that between different specimens is 0.7%. The quadratic least-

square fit to the specific heat capacity versus temperature data, in the range 1400 < T < 1900 K, is

$$c_p = 1547.32 - 1.18601\ T + 4.30405 \times 10^{-4}\ T^2 \qquad (2)$$

where c_P is in $J \cdot kg^{-1} \cdot K^{-1}$, and T is in K.

Specific heat capacity in the liquid was obtained from microsecond experiments. The enthalpy versus temperature function is linear in the liquid range yielding a constant value for the specific heat capacity. The average value of specific heat capacity in the liquid is 931 $J \cdot kg^{-1} \cdot K^{-1}$, with a standard deviation of 15 $J \cdot kg^{-1} \cdot K^{-1}$ (1.6%).

The values obtained for the specific heat capacity in the solid state from microsecond experiments agree within the expected uncertainties with the results obtained with the more accurate millisecond technique.

4.2. Electrical Resistivity

The variation of electrical resistivity as a function of temperature in the solid and liquid range is shown in Table II. The data from the millisecond experiments are presented for the solid alloy. The reproducibility of measurements for an individual specimen is 0.2% and that between different specimens 0.3%. The linear least-square fit to the electrical resistivity versus temperature data, in the range 1400 < T < 1900 K, is

$$\rho = 1.60961 + 7.68838 \times 10^{-5} T \quad (3)$$

where ρ is in μΩm , and T is in K.

Electrical resistivity in the liquid was obtained from microsecond experiments. The reproducibility of measurements between different specimens is 0.4%. The linear least-square fit to the electrical resistivity versus temperature data, in the range $2000 < T < 2300$ K, is

$$\rho = 1.89193 - 1.16055 \times 10^{-4} T \quad (4)$$

where ρ is in μΩm , and T is in K.

Considering the different thermal expansion behavior, the values obtained for the electrical resistivity in the solid state from microsecond experiments agree well with the results obtained with the more accurate millisecond technique.

4.3. Estimate of Uncertainties

A detailed analysis of sources and magnitudes of standard uncertainties in specific heat capacity and electrical resistivity measurements with the millisecond technique and the microsecond technique is given in [1, 2] and [4], respectively.

The combined (using the root-sum-of-squares method) standard uncertainties yield ±3% for expanded uncertainty (on a confidence level of 95%) for the measured values of specific heat capacity in the solid phase and ±7% in the liquid state. A value of ±1.5% for the expanded uncertainty is estimated for the electrical resistivity in the solid, and ±3% in the liquid phase.

5. DISCUSSION

The only measurements of specific heat capacity and electrical resistivity of solid 90Ti-6Al-4V near its melting range reported in the literature were performed using a similar millisecond pulse-heating technique [3]. The values for specific heat capacity obtained in our experiments agree very well (0.14% average deviation in the range 1450-1900 K), maximum deviation 0.5%) with the results of Cezairliyan et al. [3]. The average deviation of our values of electrical resistivity from values of Cezairliyan et al. in the same temperaure range is 0.12% and the maximum deviation is 0.3%.

Comparing millisecond with microsecond experiments, differences in some measured properties bigger than the expected uncertainties are noticeable in the vicinity of the (α+β)- to β-phase transition and in the melting range. This is probably due to the very high speed of the microsecond experiments, removing the experimental conditions from thermal equilibrium. The heating rate is approx. 3000 $K \cdot s^{-1}$ in millisecond experiments and 3 x 10^7 $K \cdot s^{-1}$ in microsecond experiments. Figure 1 shows the electrical resistivity as a function of specific enthalpy (related to 298K), which is proportional to temperature. The difference in electrical resistivity at higher enthalpy is, as stated before, due to

different thermal expansion behavior. But with the higher heating rate of the microsecond experiments, the β-phase with its lower electrical resistivity is reached at a higher enthalpy value.

A similar effect is noticed at melting. Figure 2 shows the radiance temperature as a function of specific enthalpy in the vicinity of melting. The melting of the specimen occurs with a temperature plateau instead of a temperature interval and the plateau is shifted towards higher temperatures.

ACKNOWLEDGMENT

This work was supported in part by the Fonds zur Förderung der wissenschaftlichen Forschung (FWF), Vienna, Austria under contract No. P12804-PHY.

REFERENCES

1. A. Cezairliyan, M. S. Morse, H. A. Berman, and C. W. Beckett, *J. Res. Natl. Bur. Stand.* **74A**:65 (1970).
2. P. Reiter and E. Kaschnitz, *High Temp.-High Press.*, in press.
3. A. Cezairliyan, J. L. McClure, and R. Taylor, *J. Res. Natl. Bur. Stand.* **81A**:251 (1977).
4. A. Cezairliyan and J. L. McClure, *Int. J. Thermophys.***8**:577 (1987).
5. J. C. De Vos, *Physica* **20**:669 (1954).
6. H. Preston-Thomas, *Metrologia* **27**:3 (1990).
7. H. Preston-Thomas, *Metrologia* **27**:107 (1990).

Table I. Chemical Composition of the 90Ti-6Al-4V Alloy in Percent (Al, V) and ppm (Others) per Mass

Material	Elements									
Form	Ti	Al	V	Fe	C	Cr	Cu	Ni	Mn	Ca
Tube	Major	6.2	4.1	2200	130	150	18	91	19	4
Wire	Major	6.4	4.3	1900	130	49	30	43	4	3

Table II. Specific Heat Capacity and Electrical Resistivity[a] of 90Ti-6Al-4V Alloy

T (K)	c_P ($J \cdot kg^{-1} \cdot K^{-1}$)	ρ (μΩm)
1400	730	1.72
1500	737	1.72
1600	752	1.73
1700	775	1.74
1800	807	1.75
1900	848	1.76
melting		
2000	931	1.66
2100	931	1.65
2200	931	1.64
2300	931	1.63

[a] based on room temperature dimensions of the specimen

FIGURE CAPTIONS

Fig. 1. Variation of electrical resistivity as a function of specific enthalpy for a typical millisecond experiment (heating rate approx. 3000 $K \cdot s^{-1}$), and a microsecond experiment (heating faster by a factor of 10,000) on 90Ti-6Al-4V specimens in the (α+β)- and β-phase.

Fig. 2. Variation of specific enthalpy as a function of radiance temperature for a typical millisecond experiment (heating rate approx. 3000 $K \cdot s^{-1}$), and a microsecond experiment (heating faster by a factor of 10,000) on 90Ti-6Al-4V specimens in the vicinity of melting.

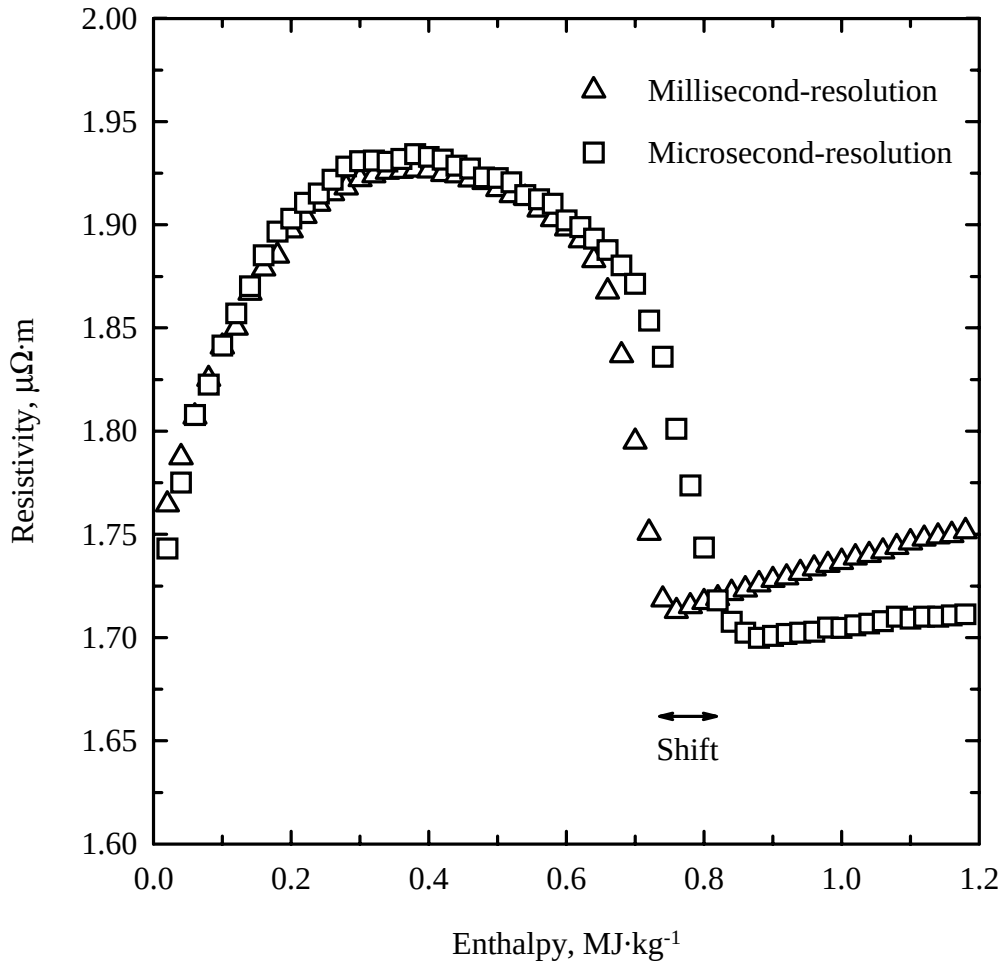
Millisecond-resolution
Microsecond-resolution
Shift
Resistivity, μΩ·m
Enthalpy, MJ·kg-1
2.00
1.95
1.90
1.85
1.80
1.75
1.70
1.65
1.60
0.0
0.2
0.4
0.6
0.8
1.0
1.2

Fig. 1.

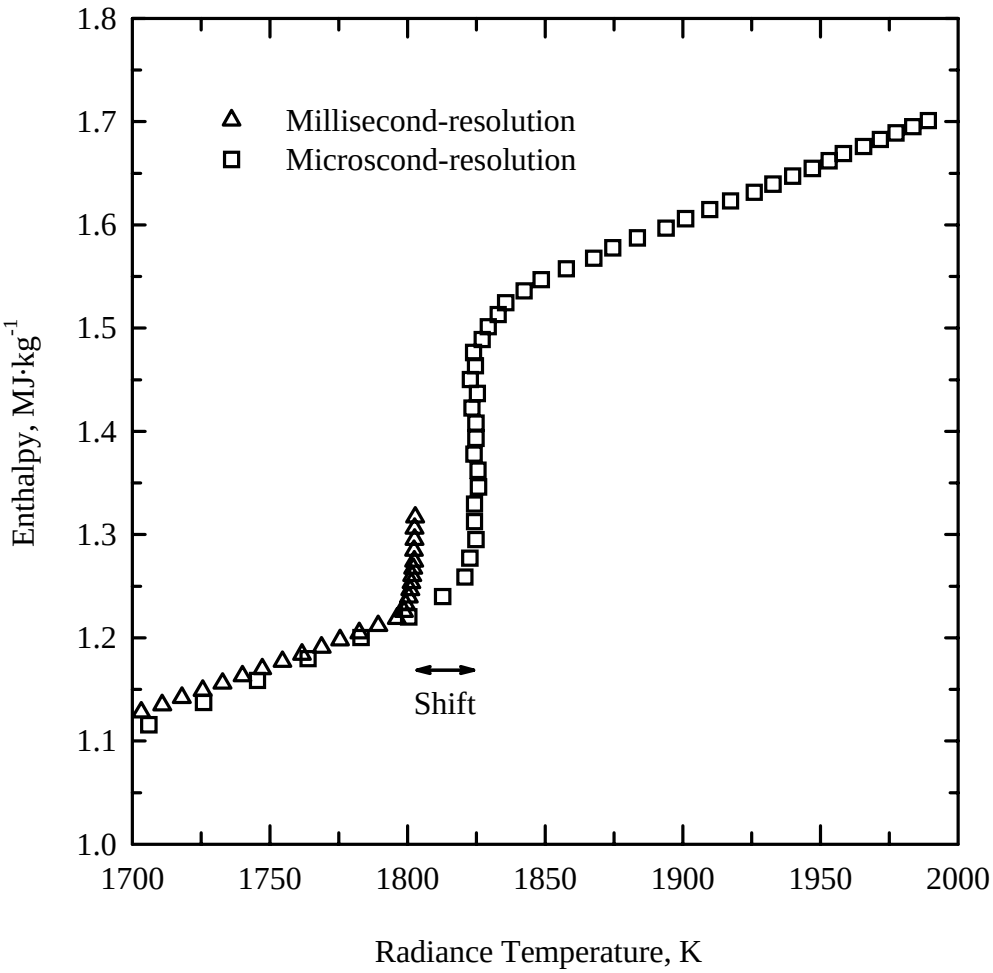
Millisecond-resolution
Microscond-resolution
Shift
Enthalpy, MJ·kg-1
Radiance Temperature, K
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1700
1750
1800
1850
1900
1950
2000

Fig 2.